# GREEN SYNTHESIS OF PURE AND DOPED MAGNESIUM OXIDE NANOPARTICLES FOR EFFECTIVE CATALYTIC, ANTIOXIDANT AND ANTIBACTERIAL APPLICATIONS

**SYNOPSIS**

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## 1. Introduction

Nanotechnology has made appreciable advances in science and technology during the past few years. Metal oxide nanoparticles have attracted the researchers particularly due to their diverse forms, sizes and high surface areas. Particles in the nanoscale differ from their bulk forms in reference to their physical, chemical, and biological characteristics (Ishak et al., 2019; Sorbiun et al., 2018). There are mainly two approaches to synthesize nanoparticles namely; top-down and bottom-up. Atoms and molecules are employed as the fundamental units in the bottom-up method to create intricate nanomaterials whereas top-down techniques focus on the reducing size of bulk particles. Based on the techniques or ingredients used in the production process, the synthesis methodologies are further divided into physical, chemical, and green or biogenic synthesis (Ishak et al., 2019; Jeevanandam et al., 2016). However, green synthesis techniques have been considered as the most affordable, safe, biocompatible and environment friendly as it does not involve any harsh or hazardous chemicals and expensive equipment for synthesis. For the biological generation of nanoparticles, a variety of environment friendly agents have been used as biocatalysts, including plant extracts, bacteria, fungi and yeasts (Fouda et al. 2021). Amongst them, plant extracts are considered as excellent source for green synthesis of nanoparticles as they contain a variety of biomolecules like flavones, terpenoids, polysaccharides and other bioactive components which play a key role in reduction and stabilization of nanoparticles. Moreover, these biomolecules enhance the surface properties of nanoparticles which have a synergistic impact on biological applications, such as antimicrobial, antioxidant, anticancer etc. (Fouda et al. 2021; Kuunal et al., 2108).

Though various metal/metal oxide nanoparticles such as Ag, Au, ZnO, CuO, $Fe_2O_3$, $TiO_2$ has been tailored by researchers by green methods, but Magnesium oxide nanoparticles (MgO NPs) distinguishes itself for its exceptional optical transparency, thermodynamic stability, corrosion resistance, biocompatibility, high surface activity, low refractive index, and high mechanical strength. In addition, it also exhibits photocatalytic, antimicrobial, antioxidant, anticancer and antifungal properties (Fouda et al. 2021; Mageshwari et al., 2013; Abinaya et al., 2021). Due to their diverse features, MgO nanoparticles have been widely used in the catalysis, hazardous waste treatment, antimicrobial agents, in bone repairing and in cancer therapy (Srivastava et al., 2015).

It has been established by various researchers that physiochemical properties of nanoparticles can also be altered by adding an impurity into the crystal structure as ‘Dopant’. Doping usually leads to alteration of pristine metal oxide’s crystal structure either by widening

or compressing the band gap, which has an impact on the overall optical, electrical, and catalytic performance of the material (Saleh et al., 2016). Both metals and non-metals can be used as dopants. The most widely used non-metal dopants are carbon, nitrogen, chlorine, phosphorus, fluorine etc. that can either act as an interstitial dopant or as an oxygen site substitute. Metals which are generally employed as dopants are first row transition metal, post transition metals or alkaline earth metals. Transition metals with $d^8$ or $d^9$ configuration when utilized as dopant introduces additional electronic states between original band gap, acting as electron traps. This prolongs the life time of $e^-$ and $h^+$ for greater catalytic performances (Medhi et al., 2020).

MgO NPs can be synthesized by various means such as, sol-gel, microwave-assisted, hydrothermal, solvothermal, wet chemical method etc. However, each of these techniques mandates the employment of expensive and environmentally hazardous reducing and capping/stabilizing agents such as polyvinyl alcohol, polyethylene glycol, and polyvinyl pyrrolidone (Ishak et al., 2019; Fouda et al., 2021). Therefore, researchers have diverted their attention towards synthesis of MgO NPs by green methods. Till now, researchers have fabricated MgO NPs via biological means by employing plant extracts of strawberry seeds (Khan et al., 2022), *Ficus religiosa* leaf (Baliyan et al., 2022), *Pisonia alba* leaf (Sharmila et al., 2019), *Ocimum sanctum* leaf (Sobana, Premlatha and Preethika, 2018), *Mucuna pruriens* L. seeds (Rahmani-Nezhad et al., 2017), *Artimesia abrotanum* plant (Dobrucka, 2018). The main advantages of using plant extracts are their ready availability and safe handling. Further, different metabolites like sugars, alkaloids, terpenoids, polyphenols, phenolic acids, proteins, etc., present in them help in reducing metal ions and act as stabilizing agents for synthesized nanoparticles (Ishak et al., 2019; Kuunal et al., 2018). Present research aims to synthesize MgO NPs by green synthetic approach by employing leaf extract of Mint, Aloe vera and Tulsi plants.

*Mentha piperita*, commonly known as 'Mint', used worldwide for its flavour, fragrance and therapeutic applications **(Figure 1a)**. Menthol, menthone, and menthyl acetate are the three primary components of the essential oil found in mint leaves. Flavonoids, including flavones and flavonones, phenolic acids like gallic acid, rosmarinic acid, vanillic acid and lignans are abundant in Mint plant. Different studies have demonstrated that mint and its compounds have a variety of biological effects, including anti-inflammatory, insecticidal, anticancer, and antibacterial characteristics (Trevisan et al., 2017; Das et al., 2021).

*Aloe barbadensis,* also known as 'Aloe vera', used worldwide for its medical and pharmacological purposes (**Figure 1b)**. The different phytochemicals present in its

mucilaginous gel are emodin, aloin, aloesin, lupeol, amylase, peroxidase, catalase, saponins, lignin, salicylic acid etc. which possess anticancer, antidiabetic and antioxidant properties (Sanchez et al., 2020).

*Ocimum sanctum*, also known as 'Tulsi', is a medicinal plant used for its antioxidant, anti-inflammatory, antimicrobial, anticancer, anti-pyretic, analgesic, antidiabetic and wound healing properties (**Figure 1c)**. The main bioactive molecules present in its leaf extract are eugenol, carvacrol, linalool, urosolic acid, rosmarinic acid, β- caryophyllene etc. (Pattanayak et al., 2010).

**Figure 1:** (a) Mint, (b) Aloe vera and (c) Tulsi

## 2. Literature review

Magnesium oxide is an inorganic metal oxide characterized by a wide band gap of 7.8eV. Its properties undergo excellent modification when reduced to nanoscale. Due to small size, large surface area and biocompatible nature of MgO NPs, they have found utility in the fields of biotechnology, catalysis, optical devices, biomedicine, and environmental remediation (Balakrishnan et al., 2020; Fernandes et al., 2020; Zwijnenburg et al., 2021). Following is a brief review of the literature on the green synthesis of MgO NPs:

In order to produce MgO NPs, Joghee et al. (2019) separated biomolecules from *Pisonia grandis R. Br.* plant leaf extract and utilized it for biosynthesis of MgO NPs. They suggested that, among the several biomolecules in the mixture, 'mome inositol' would have acted as an encapsulating agent through the chelation of $Mg^{2+}$ ions. Likewise, Khan et al. (2022) also proposed a tentative mechanism for synthesizing MgO NPs using strawberry fruit extract. They established that the phytochemical, Quercetin-3-glucuronide present in strawberry fruit formed a complex with $Mg^{2+}$ ion which finally converts to MgO NPs.

Using green extracts of *Amaranthus tricolor*, *Amaranthus blitum*, and *Andrographis paniculata* leaves, Jeevanandam et al. (2017) have synthesized MgO NPs and concluded that the stability and size of the NPs are mostly influenced by factors such as type of leaf extract, concentration of metal precursor, heating temperature, and reaction duration involved in synthesis process.

Using biocompatible *Acasia* gum, Srivastava et al. (2015) synthesised MgO nanoflowers and demonstrated that these nanoparticles may be utilized to filter synthetic waste water containing divalent metal ions as $Co^{2+,}$ $Cd^{2+}$, $Cu^{2+}$, $Zn^{2+}$, $Pb^{2+}$, $Mn^{2+}$, and $Ni^{2+}$ ions. Similarly, Fouda et al. (2021), bio-fabricated MgO NPs utilizing *Aspergillus niger* strain F1 and successfully employed them for the catalytic degradation of real textile and tannery effluent in the presence of sunlight. From FTIR studies, they claimed that fungal metabolites played an obvious role as capping and reducing agents.

According to some researchers, the phytochemicals found in plant extracts also functionalize the surface of nanoparticles with their biological characteristics, such as anti-inflammatory, anti-microbial, antioxidant, anticancer properties which tends to have a synergistic effect on the overall properties of NPs (Kuunal et al., 2018). In order to bio fabricate MgO NPs, Baliyan et al. (2022) utilized the antioxidant properties *of Ficus religiosa* and examined their capacity to scavenge free radicals, specifically 2,2-Diphenyl-1-picryl hydrazyl (DPPH) radicals. Similarly, Amrulloh et al. (2021) used *Moringa oleifera* bark extracts as bioactive agent in the synthesis of MgO NPs from $MgCl_2.6H_2O$. They obtained the particles in the size range 60-100 nm and observed that the resultant NPs have strong antibacterial and antioxidant capabilities.

Several studies have revealed that MgO nanoparticles have bactericidal properties. Due to electrostatic interactions, MgO NPs adhere to bacterial cell membranes, causing its damage and distortion resulting in leakage of intracellular contents and eventually death of bacteria (Stanic and Tanaskovic, 2020). One such study by Vergheese et al. (2018) showed that MgO NPs mediated by leaf extract of *Trigonella foenum-graecum* had substantial antibacterial activity against both gram-positive and gram-negative bacteria.

Employing *Emblica officinalis* fruit extract as a capping agent, Ramanujam and Sundrarajan (2014) synthesized MgO NPs of particle size 27 nm and coated the cotton fibers with these green-generated MgO NPs and investigated them for their antibacterial potential. The treated cotton fabric exhibited stronger antibacterial properties towards *S. aureus* than *E. coli*.

Moorthy et al. (2015) biosynthesized MgO NPs using Neem (*Azadirachta indica*) leaf extract as a reducing agent and Mg $(NO_3)_2$ as precursor and investigated the seed germination process of *Cicer arietinum* (chick pea) and *Solanum lycopersicum* (tomato). Their results have revealed that the germination process was significantly influenced by MgO NPs in *Cicer arietinum*.

Pugazhendhi et al. (2019) biosynthesized MgO NPs using marine brown algae *Sargassum wightii*. They investigated that these tailored nanoparticles possessed potent antibacterial and anti-fungal activity against human pathogens. They also exhibited significant cytotoxicity against lung cancer cells. In addition, these nanoparticles resulted in quick degradation of organic dye, methylene blue on exposure to sunlight and UV irradiation.

Hassan et al. (2021) synthesized MgO NPs utilising the metabolites present in fungal strain *Rhyzopus oryzae*. They tested these biosynthesized NPs for their antimicrobial activity as well as for decolorization of tannery effluents. They demonstrated their efficacy against *B. subtilis, S. aureus, P. aeruginosa, E. coli*, and *C. albicans* as antimicrobial agents and also reported their maximum decolorization efficiency to be 95.6% at 100 µg/100ml in 180 minutes.

Using $Mg(NO_3)_2$ as precursor and tea extract as a biological agent, Kumar et al. (2021) produced MgO NPs of particle size 40–50nm and examined their antibacterial behaviour against bacterial pathogens *E. Coli, S. aureus* and *S. pyogenes* and observed 7–10 mm zone of inhibition. Moreover, they also employed them for the photodegradation of Victoria blue dye under UV light exposure and achieved 88% degradation in 90 minutes.

Numerous studies have been conducted to examine the impact of metal dopant on the antibacterial activity of MgO NPS. In one such investigation by Cai et al. (2017), who revealed that upon doping with Ag, the size of MgO NPs decreased and more bacterial cells died due to increased production of reactive oxygen species (ROS).

Almontasser and Parveen (2022) synthesised MgO NPs doped with Fe, Co and Ni via Sol-gel method. From UV-Visible spectral and XRD analysis, they concluded that band gap increased following doping while maintaining their cubic structure. Furthermore, doping improves magnetic properties of MgO NPs and these doped MgO NPs were more effective as antibacterial agents.

By using the Sol-gel process, Rajendran et al. (2018) synthesized pure and Cu-doped MgO NPs and observed reduction in their band gap values upon doping through UV spectral investigations. Compared to their pure counterparts, Cu-doped MgO NPs demonstrated superior antibacterial activities due increased ability to generate ROS. Similarly, Rao et al. (2013) synthesized $Li^+/Zn^{2+}/Ti^{4+}$ doped MgO NPs by sol-gel process and observed the impact

of doping on the antibacterial activity against *E. coli*. They have reported that Li doped MgO NPs possessed best antibacterial activity.

Pure and Cu-doped MgO NPs were produced by Cui et al. (2014) using a chemical co-precipitation method. By using X-ray photoelectron spectroscopy (XPS), they examined the successful incorporation of $Cu^{2+}$ ions into the MgO lattice and noticed a reduction in size of NPs as the concentration of dopant increases. According to them, these doped MgO NPs had improved antibacterial properties and displayed improved chlorine adsorption.

Sharma et al. (2015) & Sharma et al. (2020) synthesized pure and Zn doped MgO NPs through a Sol-gel technique. They both have reported that the doped NPs had improved optical characteristics and could be used in a variety of UV-visible optoelectronic devices.

After carefully reviewing the literature on MgO NPs, it may be concluded that while the process of biologically synthesizing MgO NPs replaces the use of toxic chemical agents, at the same time these bioactive molecules found in plant extract can also be used as encapsulating agents to control the size of NPs. Furthermore, it has been documented in the literature that doping of MgO NPs via various chemical methods increases their optical characteristics by introducing many surface flaws, which improves their physico-chemical and biological properties. However, to best of our knowledge, there is no published literature on the 'green synthesis of doped MgO NPs, which may also result into enhanced optical, catalytic, and biological capabilities of environment friendly doped MgO NPs.

## 3. Research Gap Identification

It is well clear from the literature review that biologically synthesized MgO NPs can also be used as photocatalysts and antimicrobial material with better results. These properties make so obtained MgO as a wonderful material for environmental remediation. However, to best of our knowledge, the green methods of synthesis of MgO NPs have not been explored utilizing the medicinal plant extracts of *Mentha piperita*, *Aloe barbadensis*, and *Ocimum sanctum* plants. Moreover, green synthesis and the control of various properties via Co/ Cu/Zn doping have not yet been done in conjunction for obtaining MgO NPs. Therefore, it would be an innovative approach to biologically synthesize and characterize these undoped/ doped (Co/Cu/Zn) MgO NPs for effective photocatalytic, antibacterial and antioxidant applications.

## 4. Objectives

In light of the aforementioned discussions, the following are the aims of the current study:

1. To green synthesize pure and doped MgO NPs using Co, Cu and Zn as dopants.
2. To characterize the synthesized nanoparticles by using different characterization techniques like XRD (X-ray diffraction), SEM (Scanning electron microscopy), EDX (Energy dispersive X-ray), TEM (Transmission electron microscopy), UV-Visible, and FTIR (Fourier transform infrared) spectroscopy.
3. To investigate the photocatalytic, antioxidant and antibacterial applications of synthesised nanoparticles.

## 5. Proposed Methodology

In the current research, we emphasized on the green synthesis of pure and Co/Cu/Zn doped MgO NPs using three medicinally significant plants, namely Mint, Aloe vera, and Tulsi.

### 5.1 Materials

Magnesium nitrate hexahydrate, Cobalt nitrate hexahydrate, Cupric nitrate trihydrate, Zinc nitrate hexahydrate, Sodium hydroxide, 99% ethanol, Methyl orange, Malachite green, $H_2O_2$, Mueller Hinton Broth, Agar-agar type-I, Ascorbic acid and 2,2-diphenyl-2-picryl hydrazyl hydrate (DPPH) are the chemicals used in this experimental work. Bacterial strains *Bacillus subtilis* (MTCC 441), *Staphylococcus aureus* (MTCC 737), and *Escherichia coli* (MTCC 739) are used in this study. Plant leaves of Aloe Vera (*Aloe barbadensis*), Tulsi (*Ocimum sanctum*) and Mint (*Mentha piperita*) are collected from nearby areas for preparation of extracts.

### 5.2 Methods

#### 5.2.1 Preparation of leaf extract

Green leaves of Mint, Aloe Vera and Tulsi are collected and rinsed 2-3 times with tap water and then with distilled water to get rid of any impurities. About 10 g of leaves are minced and refluxed in 100 ml of distilled water for about 30 minutes. The refluxed extract is filtered by using Whatman filter paper and used as a leaf extract for the synthesis of MgO NPs.

#### 5.2.2 Synthesis of MgO Nanoparticles

For the synthesis of MgO NPs, 100 ml of 0.05 M Magnesium nitrate, $Mg(NO_3)_2.6H_2O$ solution to be taken in a beaker. To this solution, 10 ml of leaf extract (Mint/ Aloe Vera/ Tulsi) to be added dropwise and stirred magnetically at 70 $^0$C. Sodium hydroxide, NaOH solution will

be used to adjust pH in the range 11-12. The reaction mixture to be stirred magnetically for 3 hours at 70 $^0$C. The reaction will be monitored with the help of color change of the reaction mixture. MgO NPs will be obtained as precipitate (**Figure 2**). For preparing Co/Cu/Zn doped MgO NPs, 100ml of 0.045 M $Mg(NO_3)_2.6H_2O$, 100 ml of 0.005 M of dopant ($Co(NO_3)_2$/$Cu(NO_3)_2$/$Zn(NO_3)_2$) solution and 20 ml of leaf extract will be taken. The pure and doped MgO NPs obtained will be washed three time with distilled water and then with ethanol to remove excess of $Mg(NO_3)_2$ and plant material. MgO NPs to be dried for 24 hours at 60 $^0$C and calcined at 500 $^0$C for 2 hours. The dried material will then be crushed with a mortar and pestle.

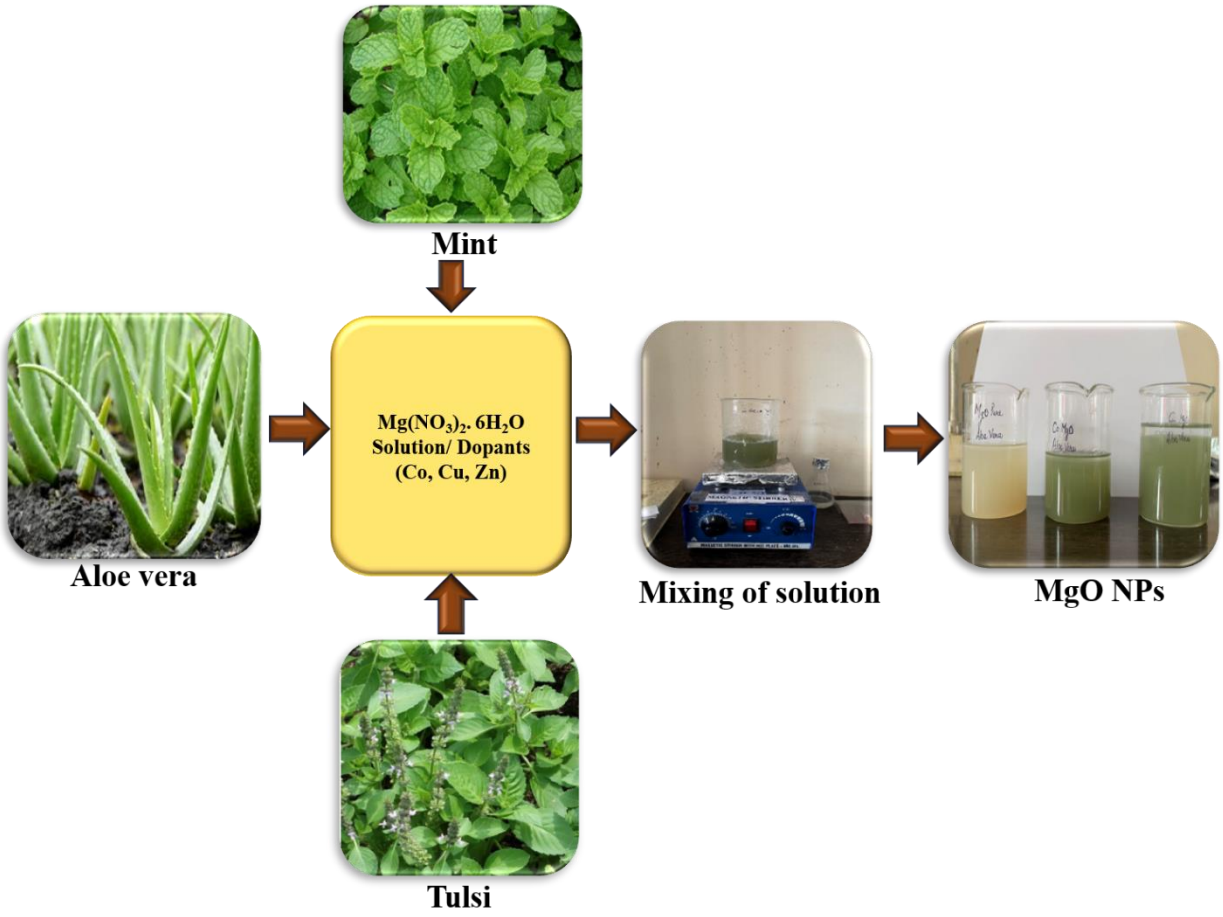


**Figure 2:** Green synthesis of MgO nanoparticles

### 5.3 Characterizations

The characterization of synthesized samples will be done by using XRD, UV-Visible, FTIR, SEM, EDX and TEM to confirm the formation of MgO NPs, average crystallite size,

phase composition, nature of bonds and functional groups, surface morphology, elemental composition and average particle size.

### 5.4 Applications

#### 5.4.1 Photocatalytic activity

In this study, the green synthesized pure and doped MgO NPs will be used for the degradation of dyes; Methyl orange and Malachite green. The photodegradation rate will be evaluated by measuring the absorbance by UV-Visible spectrophotometer. The procedure will be adopted as mentioned by Mageshwari et al., 2013 and Sharma et al., 2021.

#### 5.4.2 Antioxidant activity

The antioxidant activity of synthesized MgO NPs will be examined by investigating the radical scavenging ability against 2,2-dipenyl-1-phenyl hydrazyl (DPPH) free radical at different concentration. The absorbance of solutions will be measured by UV-Visible spectrophotometer. The method will be followed as given by Ammulu et al., 2021.

#### 5.4.3 Antibacterial activity

The synthesized nanoparticles will be assessed for their antibacterial activity by investigating the zone of inhibition formed against three bacterial pathogens namely; *Bacillus subtilis* (MTCC 441), *Staphylococcus aureus* (MTCC 737), and *Escherichia coli* (MTCC 739). The procedure will be followed as given by Sharma et al., 2021.

## 6. Thesis Outline

**CHAPTER 1 (Introduction)**

Chapter 1 will include introduction to latest development in nanomaterials, different methods to synthesize metal oxide and doped metal oxide nanoparticles, biological methods, plant extracts, characterization, properties and various applications by reviewing the latest literature.

**CHAPTER 2 (Literature Review)**

This chapter will deal with intense literature survey of synthesis and applications of metal oxide nanoparticles. This chapter will also include research gap identification and objectives of proposed study.

**CHAPTER 3 (Materials and Methods)**

It includes the experimental details on material used, biological methods of synthesis, characterization techniques and applications of synthesized nanoparticles.

**CHAPTER 4 (Results and Discussion)**

This chapter will include detailed discussion on results of present work which includes the key points and various aspects of synthesis, characterization and applications of synthesized NPs.

**CHAPTER 5 (Summary and Conclusions)**

This chapter will summarise and correlate all the above-mentioned chapters in lucid manner.

**CHAPTER 6 (References)**

This chapter will include the all references.

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**CERTIFICATE**
**(SYNOPSIS APPROVAL CERTIFICATE)**

This is to certify that the Synopsis for Ph.D. Thesis titled, **"GREEN SYNTHESIS OF PURE AND DOPED MAGNESIUM OXIDE NANOPARTICLES FOR EFFECTIVE CATALYTIC, ANTIOXIDANT AND ANTIBACTERIAL PROPERTIES"**, submitted by **Ms. Dimpal Chauhan** to the Career Point University, Hamirpur (H.P.), India in partial fulfillment of the requirements for the degree of **DOCTOR OF PHILOSOPHY** in the Department of **Chemistry**, School of **Basic and Applied Sciences** has been approved by the Research Advisory Committee (RAC) after an Oral Examination of the student on dated 20.02.2021.

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